

**CHARACTERIZATION AND PROPERTIES OF
PALM KERNEL SHELL REINFORCED
POLYESTER COMPOSITES**

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UNIVERSITI MALAYSIA PERLIS

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CHARACTERIZATION AND PROPERTIES OF PALM KERNEL SHELL REINFORCED POLYESTER COMPOSITES

by

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APPROVAL AND DECLARATION SHEET

This project report entitled “Characterization and Properties of Palm Kernel Shell Reinforced Polyester Composites” was prepared and submitted by Nor Mazlina binti Abdul Wahab (Matrix No: 0831620293) and has been found satisfactory in term of scope, quality and presentation as partial fulfillment of the requirement for the Master of Science Polymer Engineering, School of Materials Engineering in Universiti Malaysia Perlis (UniMAP).

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PENCIRIAN DAN SIFAT-SIFAT KOMPOSIT POLIESTER TAK TEPU DIPERKUAT TEMPURUNG KELAPA SAWIT

ABSTRAK

Komposit poliester tak tepu (UP)/tempurung kelapa sawit (PKS) telah disediakan dengan kaedah pelapisan tangan. Kesan pembebanan pengisi di dalam komposit UP/PKS ke atas sifat-sifat mekanikal dan morfologi telah dikaji. Ujian tensil dan kelenturan dilakukan terhadap komposit untuk mengetahui kesan kandungan pengisi terhadap sifat mekanikal. Keputusan menunjukkan dengan meningkatnya pembebanan PKS telah mengurangkan nilai kekuatan tensil dan kelenturan, tetapi modulus tensil dan modulus kelenturan didapati meningkat. Agen pengganding, 3-APE digunakan untuk meningkatkan sifat-sifat mekanikal komposit. Kehadiran 3-APE telah meningkatkan kekuatan tensil, kekuatan kelenturan, modulus tensil dan modulus kelenturan. Kajian mikroskop elektron pensakanan (SEM) menunjukkan bahawa interaksi antara muka pengisi-matrik telah meningkat dengan penambahan 3-APE. Kesan modifikasi kimia PKS dengan asid akrilik (AA) pada komposit UP/PKS juga telah meningkatkan kekuatan tensil, kekuatan kelenturan, modulus tensil dan modulus kelenturan. Mikrograf SEM menunjukkan komposit rawatan dengan AA mempunyai penyebaran pengisi yang lebih baik di dalam matrik poliester tak tepu. Spektra FTIR menunjukkan interaksi di antara PKS dan matrik meningkat dengan penambahan agen pengganding.

CHARACTERIZATION AND PROPERTIES OF PALM KERNEL SHELL REINFORCED POLYESTER COMPOSITES

ABSTRACT

Composites of unsaturated polyester (UP)/palm kernel shell (PKS) have been prepared by hand lay-up technique. The effect of filler loading of UP/PKS composites on mechanical properties and morphology were studied. Tensile and flexural tests have been done to determine the effect of filler loading on mechanical properties. The results showed the increased of PKS filler loading have decreased the value of tensile and flexural strengths, but increased in tensile modulus and flexural modulus. A coupling agent (3-APE) was used to improve the mechanical properties of composites. The presence of 3-APE improved the tensile strength, flexural strength, tensile modulus and flexural modulus. The study of scanning electron microscope (SEM) shows that the filler-matrix interaction was improved with incorporation of 3-APE. The effect of chemical modification of PKS with acrylic acid (AA) in UP/PKS composites increased the tensile strength, flexural strength, tensile modulus and flexural modulus. The SEM micrograph showed the treated composites with AA has better dispersion in UP matrix. FTIR spectra show the interaction between PKS and matrix was enhanced with addition of coupling agent.

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LIST OF SYMBOLS, ABBREVIATIONS OR NOMENCLATURE

UP	Unsaturated Polyester
PKS	Palm Kernel Shell
MEKP	Metyl ethyl ketone peroxide
BPO	Benzoyl peroxide
AA	Acrylic acid
3-APE	3-Aminopropyltriethoxysilane
NaOH	Sodium hydroxide
SEM	Scanning electron microscope
PP	Polypropylene
HDPE	High density polyethylene
LDPE	Low density polyethylene
MAN	Maleic Anhydride
FTIR	Fourier transform infrared
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetry
DMA	Dynamic Mechanical Analysis
ASTM	American Society for Testing and Material
UV	Ultra violet
MPa	Mega Pascal
s	Second
min	Minutes

β	Beta
g	gram
μ	Micro
μm	Micrometer
IR	Infra Red
$^{\circ}\text{C}$	Degree Celsius
$^{\circ}\text{F}$	Fahrenheit
%	Percent
wt	Weight
OH	Hydroxyl
C	Carbon
O	Oxygen
php	per hundred polymer
cm^3	Cubic centimeter
m^3	Cubic meter
mm	Millimeter
kg	Kilogram

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Nowadays, polymer composites play an important role in polymer industries and engineers always keep on searching the best material to suit with certain design and product requirement. Since the environmental awareness is growing significantly, one of the important factors to be considered in recent development of high performance material is ecological friendly with minimum effect on environment and by using renewable and recycles resources. Consequently, in recent years many researchers have been investigate on these types of materials as potential filler (reinforcing or non-reinforcing) in polymer composite system.

Some researchers had found that with some modification, these kinds of materials can give an excellent performance especially on strength and stiffness (Sangthong et al., 2009; Vilay et al., 2007; Pothan et al., 2006; Nair et al., 2001; Saha et al., 1999). Hence, there are needs on study the polymer composite by incorporation various types of fillers, especially natural fillers in the development of high performance polymer composites. Furthermore, a polymer composite is relatively easy to prepare based on the existing technology with a balanced properties as required.

Composite materials are those that are formed by the combining of two or more materials to achieve properties that are superior to those of their constituents (Wang et al., 2003). Polymer composites are one of the most rapidly developing and advanced composite materials. Polymer composites consist of a polymer resin as a matrix, with fibers as the reinforcement medium (Callister, 2007). Most of the commercially available polymers used as composite matrices are currently petroleum-based, non-biodegradable and regarded as non-sustainable material (Mohanty et al., 2000; Lu et al., 2003; Toriz et al., 2003; Wibiwo et al., 2004).

Fibrous polymer composites materials have three components i.e. fibers, matrix and interface (interphase) (Bascom, 1990-1991). The mechanical properties of composites are controlled by the strength and the elastic properties of the fibers, the matrix and the fiber-matrix bond which governs the stress transfer (Sham et al., 1997; Mahiou & Beakou, 1998). If there is weaker adhesion at the interface, the stress transfer between the fiber and the matrix is reduced (Mahiou & Beakou, 1998).

Unsaturated polyesters are extremely versatile in properties and applications and have been a popular thermoset used as the polymer matrix in composites (Callister, 2002; Aziz et al., 2005). This matrix has been used for many years in broad technology fields such as naval construction, offshore applications, waterlines and building construction (Manfredi et al., 2006). Unsaturated polyester has many advantages compared to other thermosetting resin i.e room temperature cure capability, good mechanical properties and transparency. The reinforcement of polyester with various cellulosic fibres has been widely reported (Aziz et al., 2005). Sreekumar et al., (2007) studied the mechanical properties of sisal fibre reinforced polyester composites. They observed the tensile and flexural behaviors of the sisal fiber/polyester composites as a function of fiber length and fiber loading. The surface

characteristics of natural fiber also investigated in order to improve the adhesion of the natural fiber/polymer composite (Santhong et al., 2009).

Glass, Carbon, Boron and Kevlar fibres are being used as reinforcing materials in fibre reinforced plastics (FRP) due to their high specific modulus, high stiffness to weight ratio and high strength to weight ratio compared to conventional materials. However, the materials are expensive and their use is justified only in aerospace applications. Cellulosic fibers, like henequen, sisal, coconut fiber (coir), jute, palm, bamboo, paper in their condition, as well as, several waste cellulosic products such as shell flour, wood flour and pulp have been used as reinforcement agents of different thermosetting and thermoplastic resins (Belmares et al., 1981; Cruz-Ramoz et al., 1982; Casaurang-Martinez et al., 1990 and 1991; Jain et al., 1992; Varghese et al., 1994; Ahlblad et al., 1994; Geethamma et al., 1995). Therefore, the interest in natural fibers reinforced polymer composites is growing rapidly due to the high performance in mechanical properties, significant processing advantages, low cost and density (Saheb & Jog, 1999; Mishra et al., 2004; Dweib et al., 2004).

Natural fibers have recently attracted the attention of scientists and technologists because of the advantages that these fibers provide over conventional reinforcement materials, and the development of natural fiber composites has been a subject of interest for the past few years (Scheider et al., 1995; Colberg & Sauerbier, 1997). These natural fibers are low cost fibers with low density and high specific properties. The special characteristics offered by natural fiber are biodegradable and nonabrasive, unlike other reinforcing fibers. They are also readily available and their specific properties are comparable to those of other fibers used for reinforcement. However, certain drawbacks such as incompatibility with hydrophobic polymer matrix, the tendency to form aggregates during processing and poor resistance to moisture greatly reduce the potential of natural fibers to be used as reinforcement in polymers (Saheb &

Jog, 1999). Mechanical properties of natural fibers, especially flex, hemp, jute and sisal are very good and may compete with glass fiber in specific strength and modulus (Li et al., 2007).

Among all these natural fiber reinforcing materials, jute appears to be a promising material because it is relatively inexpensive and commercially available in the required form. It has higher strength and modulus than plastics (Shah & Lakkad, 1981) and is a good substitute for conventional fibers in many situations. Bagasse fibers are getting acceptance from many researchers and it can be used as an alternative reinforcement in composite materials as well.

The performance of fiber-reinforced environmentally friendly materials are depends on the development of coherent interfacial bonding between the fibers and matrix (Piggott et al., 1986; Piggott, 1980; Drzal et al., 1993). To produce reactive hydroxyl groups and the rough surface for adhesion with polymeric material, plants fiber need to undergo physical and/or chemical treatment to modify the surface and structure (Yoldas, 2009). Alkali treatment of natural fibers was used to improve the adhesion with a polymer matrix by a number of researchers (Gomes et al., 2007; Rajulu et al., 2003; Razera & Frollini, 2004). During alkali treatment, physical structure of the fibers changes as a result of alkali's bleaching action which removes waxy materials and impurities. This action often leads to improvement of the interfacial bonding between fibers and matrix (Gomes et al., 2007). Coupling agent like silane as well plays an important role in order to improve adhesion between natural fibers and matrix such as epoxy and polyester. The influence of chemical modification of natural fibers with an organosilane coupling agent was found out by Valadez-Gonzalez et al. (1999a).

Some of the potential applications of natural fiber reinforced composites include door and instrument panels, package trays, glove boxes, arm rest and seat back (Li et al., 2004). However, their high level of moisture absorption, poor wettability by non-polar plastics and insufficient adhesion between untreated fibers and the polymer matrix can lead to debonding with age (Cao et al., 2006; Mohanty et al., 2001; Valadez-Gonzales et al., 1999b). The careful choice of the fiber and matrix pair, the fabrication method and manufacturing procedures, the final application envisaged and the fiber surface treatment is increasing the applications of natural fiber reinforced composites (Sousa et al., 2004).

Yoldas , (2009) has reported on innovative multifunctional siloxane treatment of jute fiber surface and its effect on the mechanical properties of jute/ thermoset composites. This study shows that oligomeric siloxane treatment results in an increase in tensile strength as well as flexural strength. Surface treatment of jute fiber caused a significant increase in the interlaminar shear strength (ILSS) of the thermoset composites as well. From SEM observations, jute/ thermoset composites had shown a better adhesion in the presence of oligomeric siloxane.

The other researcher has reported on the mechanical property improvement of unsaturated polyester composite reinforced with admicellar-treated sisal fibers (Sangthong, 2009). The author reported that PMMA film coating on sisal fiber surface by admicellar polymerization leads to improved mechanical properties of the sisal fiber/unsaturated polyester composite due to the improvement of the interfacial adhesion of the composite.

Other work reported on the effect of fiber surface treatment and fiber loading on the properties of bagasse fiber-reinforced unsaturated polyester composites (Vilay, 2007). In this work, the chemical treatment using sodium hydroxide (NaOH) and acrylic

acid (AA) were carried out to modify the fiber properties. Based from the researchers previous work (Vilay, 2006), the parameters for NaOH and AA treatments on the bagasse fibers were determined and vacuum bagging process was carried out to fabricate the composite system. The results show that the addition of higher amount of fiber give higher tensile and flexural properties of the bagasse fiber-reinforced polyester composites with significant trend shown by AA treated fiber based composites than untreated fiber based composites.

Due to the wide application of polymer composites nowadays, therefore it is very promising effort to study on the utilization of local and cheap renewable sources of fiber available in our country for the production of polymer composite. In this research, the influence of filler loading and the effect of chemical modification of unsaturated polyester/palm kernel shell composite on mechanical properties and morphology have been studied.

1.2 Objectives of Study

The primary objective of this study is to characterize and determine the properties of unsaturated polyester (UP)/palm kernel shell (PKS) composites on a different composition of PKS loading with an aim of developing a new natural filler material as useful filler in composites based on thermoset resin. Several different compositions of PKS loading in UP/PKS composites were prepared in order to study;

1. the effect of palm kernel shell (PKS) as a filler on mechanical properties, morphology and FTIR of UP/PKS composites.
2. the effect of a silane coupling agent (3-aminopropyltriethoxysilane) on mechanical properties, morphology and FTIR of UP/PKS composites.
3. the effect of Acrylic Acid on mechanical properties, morphology and FTIR of UP/PKS composites.

In this research, there a few significant interest in order to characterize and investigate the properties of UP/PKS composites which include tensile properties, flexural properties, morphology and FTIR. The tensile test and flexural test were done to determine the effect of PKS content of UP/PKS composites. Scanning electron microscope of tensile fracture surface of the UP/PKS composites was done to study filler dispersion, adhesion and interfacial interaction between filler and matrix and to detect the presence of microdefects.

CHAPTER 2

LITERATURE REVIEW

2.1 Composites

A composite material can be defined as a macroscopic combination of two or more distinct materials, having a recognizable interface between them and is better (stronger, tougher, and/or more durable) than each would be separately (Swanson, 1997). However, since composites are usually used for their structural properties, the definition can be restricted to include only those materials that contain reinforcement (such as fibers or particles) supported by binder (matrix) material (Hollaway, 1993). Therefore, composites typically have a discontinuous fiber or particle phase that is stiffer and stronger than the continuous matrix phase. In order to provide reinforcement, there generally must be substantial volume fraction (10% or more) of the discontinuous phase (Hollaway, 1993).

The main concept of composite is that it contains matrix materials. Typically, composite material is formed by reinforcing fibers in a matrix resin. The reinforcements can be fibers, particulates or whiskers, and the matrix materials can be metals, plastics or ceramics. The fibers can be continuous, long or short. Composites made with a polymer matrix have become more common and are widely used in various industries which can be thermoplastic or thermoset resins (Sanjay, 2002).

The concept of composites was not invented by human beings; it is found in nature. An example is wood, which is composite of cellulose fibers in a matrix of natural glue called lignin. The shell of invertebrates, such as snails and oysters, is an example of a composite. Such shells are stronger and tougher than man-made advanced composites. Scientist has found that the fibers taken from a spider's web are stronger than synthetic fibers. In India, Greece and other countries, husks or straw mixed with clay have been used to build houses for several hundred years. Mixing husk or sawdust in a clay is an example of a particulate composite and mixing straws in clay is an example of short-fiber composite.

The reinforcing fiber or fabric provides strength and stiffness to the composites, whereas the matrix gives rigidity and environmental resistance (Sanjay, 2002). The properties are strongly depending on the way the fibers are laid in the composite. The important thing to remember about composites is that the fiber carries the load and its strength is greatest along the axis of the fiber. Long continuous fibers in the direction of the load result in a composite with properties far exceeding the matrix resin itself. The same material chopped into short length yields lower properties than continuous fibers.

The properties of composite materials are determined by the properties of the components, the shape of the filler phase, the morphology of the system and the nature of the interface between the phases. Thus a great variety of properties can be obtained with composites just by alteration of one of these items (example, the morphological or interface properties). An important property of the interface that can greatly affect mechanical behavior is the strength of the adhesive bond between the phases (Nielsen & Landel, 1994). Many research works have been carried out to identify the parameters that govern mechanical behavior of particulate composites. Generally, it has been found that the reinforcement effect increases with decreasing particle size and with increasing adhesion to the matrix (Marcivoch et al., 1998).